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ANALYSIS OF GAS MIXTURES BY DETERMINATION OF HEAT CONDUCTIVITY

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[Figures referred to are appended.]

In the analysis of some gases a number of difficulties arise which are conditioned by the gases' inertness. As a result, chemical methods of analysis are generally unsuitable. Analysis is usually conducted by physicochemical or physical methods based on the diversity of the characteristic physical properties of these gases, e.g., heat conductivity, density, adsorption on solid adsorbents, ignition potentials, etc.

It is impossible to select one physical property as a basis for a universal analysis procedure applicable to any binary mixture with any ratio of components. In each individual case, that method must be selected which will give optimum results with respect to sensitivity.

The fundamental shortcoming of many physical methods of gas-mixture analysis, particularly those methods based on the determination of heat conductivity, saturated vapor pressure, density, etc, lies in the fact that these methods are suitable only for quantitative analysis of a binary mixture of known qualitative composition.

The present article describes a device for the analysis of gas mixtures by determination of their heat conductivity. The device was built and tested by the authors in the rare-gases laboratory of the All-Union Electrical Engineering Institute. The apparatus is very convenient, permitting analysis in 2-3 minutes, and is suitable for continuous operation with automatic recording of results. Gas consumption for analysis does not exceed 100 to 200 ml. If so desired, all the gas may be collected in a gas holder, thus reducing losses to zero. During continuous analysis, the rate of passage of the gas through the apparatus is 40-50 ml/min. The analyzed gas may then be joined with the main gas flow.

To analyze a gas mixture on the basis of heat conductivity, the usual procedure is to measure the resistance of an electrically heated metal filament placed in the gas medium. The temperature of this filament, and consequently its resistance, depends on the heat conductivity of the surrounding medium, i.e., on the nature and composition of the gas being analyzed. Resistance is measured with a bridge, which entails rigid requirements for stabilization of the feed voltage, since the current in the diagonals increases in proportion to the cube of the applied voltage.

Figure 1 shows the analyzer circuit, including transformer Tr, fed via switch S from a 110/220-v, ac circuit. The selenium rectifier R, mounted according to the Graetz circuit, supplies voltage to potentiometer P over ballast resistor B which is series-connected. Secondary transformer voltage is 15 v. The ballast resistor characteristic is 3.5-6 v and 1.25 a, full potentiometer resistance is 3.2 ohms. Thus, the maximum voltage which may be applied to the measuring bridge is 4 v; however, the voltage may be decreased in one-volt intervals.

Experience has shown this method of voltage stabilization to be entirely satisfactory for ordinary operations.

If simplification of the circuit is desired, the transformer, rectifier, and ballast resistor may be eliminated and replaced by a suitable 4- to 6-v storage battery. It is recommended in this case that the potentiometer resistance be increased to 40 ohms and that a rheostat with a resistance of the order of 40 ohms and a milliammeter for up to 500 ma be included in series in the battery circuit.

The measuring bridge consists of four platinum filaments with a diameter of 50 microns, 200 mm long, two of which (H_1) are in contact with the test gas, the other two, (H_2) being in contact with a control gas. Since in the process of assembly, the resistances of the filaments may not be perfectly matched and, because of wear in prolonged use, the resistance will vary somewhat, the matching rheostats rh_1 and rh_2 are included in the circuit to allow balancing of the bridge by reducing the galvanometer setting to zero. The resistance of one filament is of the order of 11.6 ohms; the resistance of a rheostat is about 0.5 ohm.

A mirror galvanometer with a sensitivity of $2.4 \cdot 10^{-5}$ a per graduation is connected into the bridge diagonal.

The measuring bridge is mounted in a heavy brass cylinder 50 mm in diameter and 220 mm high. Four highly accurate symmetrical channels 4 mm in diameter and a fifth, central channel 6 mm in diameter are drilled in the cylinder. Two of the peripheral channels are connected with the central channel by holes drilled radially in the cylinder. The other two channels are joined together by tubes running outside of the cylinder, and carry the comparison gas.

A schematic drawing of the body of the analyzer is shown in Figure 2. The filaments, 1, are soldered at their upper ends to platinum rods, 2, which are soldered into glass caps, 3. The caps are fastened securely and sealed. The lower ends of the filaments are fastened to molybdenum springs, 4, which connect with the end of a bolt, 5, for tightening the filaments. The bottom of the cylinder around the channels is coated with a heat-resistant lacquer to prevent electrical contact between the bolts, 5, and nuts, 6, and the cylinder.

The rate of gas flow through the channels is measured by rheometers; phosphoric anhydride cartridges are installed to dry the gases. It is obvious that the greater the difference in heat conductivity of the gases in the mixture, the greater the sensitivity of the apparatus.

As a rule, the heat conductivity of a gas mixture varies with the change in molar concentrations of the components according to the principle of additivity. However, there are many known exceptions to this rule. And in addition, heat transfer from the filament into the surrounding medium by way of radiation is unavoidable because of the convection of gases in the channels and other similar phenomena. All these factors make the filament-cooling process within the apparatus extremely complex, and make it practically impossible to determine the composition of the gas by way of computation. It is necessary first to calibrate the apparatus with mixtures of known composition. These curves may be used later, provided that all conditions of electrical supply, flow rate of the gases, and sensitivity of the apparatus are precisely the same as when calibration was conducted.

The comparison chamber must be filled with gas with a heat conductivity close to that of the test gas. Ordinarily, the comparison chamber is filled with the predominant component in the test gas.

In our analysis experiments with various mixtures, the comparison chamber was filled with a readily available gas--dry nitrogen or air--and gave entirely uniform results. The entrance aperture of the comparison chamber is connected with the outlet tube, and the gas circulates only from natural convection.

Mixtures for calibration were collected in a 500-ml glass burette equipped with two cocks. The burette is filled with distilled water and the content of the components in the mixture is determined by the volume of water displaced by the gas, with subsequent conversion to the pressure of the gases in the gas holders. With this method of making up the mixtures, their calculated composition does not vary from the true figure by more than 0.1%.

The results of analyzing several binary gas mixtures by the method of measuring their heat conductivity are given below.

All calibrations were conducted with a voltage on the bridge of 4 v.

Argon-Nitrogen Mixture

Despite the fact that there is comparatively little difference between the heat conductivities of argon and nitrogen, the apparatus proved sufficiently sensitive to analyze this mixture. Figure 3 shows the calibration curves for the argon-nitrogen mixture. Curve I corresponds to maximum galvanometer sensitivity and permits determination of the content of argon in the nitrogen -- not over 40-42% -- by volume. Such a mixture corresponds to the full scale of the galvanometer at maximum sensitivity. In this instance, one graduation corresponds to 0.4% argon, based on an average over the entire scale. Accuracy of analysis is of the order of 0.2%, as confirmed by control analyses with precise stabilization of the current in the bridge.

With argon contents in excess of 40%, the galvanometer needle passes the final graduation on the scale. It becomes necessary, in this case, to reduce the galvanometer's sentivity by means of a shunt Curve II shows the result of calibration for such mixtures. The accuracy of analysis is less here, but does not fall below 0.4-0.5% argon by volume. The sensitivity of the analyzer remains constant over the entire range of measurements, i.e., up to 100% argon content. This is indicated by the rectilinearity of the calibration curves.

The great facility of this method of analyzing the given mixture is illustrated by the fact that, as a result of the near equality in heat conductivity between nitrogen and oxygen, the presence of oxygen does not interfere with the analysis of argon. It is therefore possible to make a direct analysis for argon content in an argon-nitrogen-oxygen mixture, which is a possibility with great practical interest.

Hydrogen-Nitrogen Mixture

The great difference between the heat-conductivity coefficients of these two gases requires a high degree of analyzer sensitivity for work with this mixture. A characteristic property of its calibrating curve is the convexity in the 0-100% range of hydrogen concentration (Figure 4). Consequently, the analyzer's sensitivity in the area of small concentrations of hydrogen in nitrogen is increased, while sensitivity for large concentrations of hydrogen in nitrogen is somewhat reduced. Figure 3 shows the calibration curve for mixtures with a hydrogen content up to 7%.

The sensitivity of the analyzer for such mixtures is very high -- about 17 graduations for 1% [illegible] analysis error, in practice, is governed by the accuracy with which the mixtures are made up and by the reproducibility of all the [illegible]'s operating conditions during its calibration.

Helium-Nitrogen Mixture

The difference between the heat-conductivity coefficients for helium and nitrogen is very great. This mixture is analogous in many ways to the hydrogen-nitrogen mixture. The calibrating curve was obtained for compositions of up to 13% helium in nitrogen is shown in Figure 3. Sensitivity for the analysis of these mixtures is quite high -- about nine galvanometer graduations for 1% helium.

The possibility of an analysis of helium in a helium-nitrogen-oxygen mixture is significant.

Hydrogen-Argon Mixture

The calibraing curve for this mixture is shown in Figure 5. The 6.1-ma maximum galvanometer current corresponds to pure argon. With increased hydrogen content, the current in the galvanometer drops. The maximum analyzable hydrogen content therefore corresponds to zero on the galvanometer and cannot exceed 10%.

The sensitivity of the analyzer for this mixture is also very high, reaching 11 graduations for 1% hydrogen in argon.

/Appended figures follow./

Figure 1

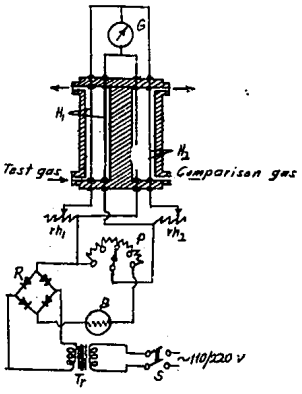


Figure 2

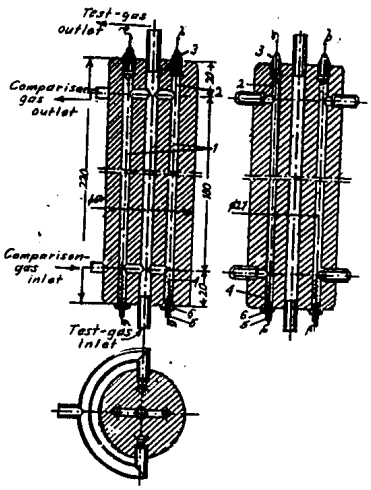


Figure 3

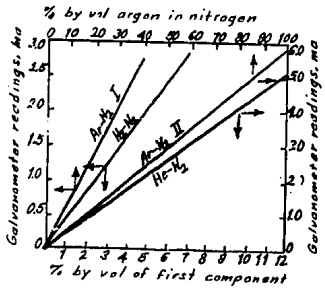


Figure 4

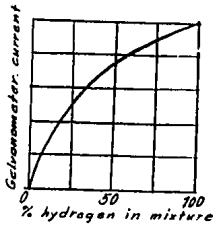
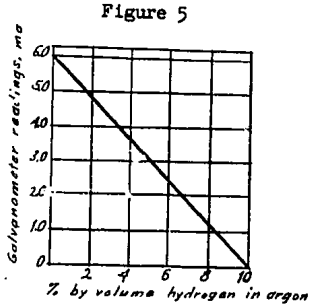


Figure 5



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